MSL954005

Prepare Mineral Samples for Analysis

Assessment & Training Manual OAB97

Science Resources & Environment

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BDV3

Certificate IV in Laboratory Techniques

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SECTION A – MINERALOGY

Exercise 1: Identification of Sulphides

Introduction

Mineralogy is a subject of geology specializing in the scientific study of chemistry, crystal structure, and physical (including optical) properties of minerals. Specific studies within mineralogy include the processes of mineral origin and formation, classification of minerals, their geographical distribution, as well as their utilization. (Wikipedia definition).

Samples in assaying terms can be categorized into three classifications – class 1 (neutral), class 2 (sulphides) and class 3 (oxides). These categories can be extended further but is not required for the purposes of this exercise.

In this exercise, some samples will be tested for the presence of sulphides using concentrated nitric acid. This test is called the ‘Nitric acid test’ and serves as a convenient method to quickly determine if sulphides are present in a sample prior to analysis.

Elements of Competency

The following elements of competency are addressed in this exercise:

- Obtain the samples
- Prepare sample for testing
- Dispose of waste and spent samples
- Maintain a safe work environment

ORE CLASSIFICATION TABLE

Reducing Samples — Neutral Samples — Oxidizing Samples

BASIC SULPHIDES — **ACIDIC** — **BASIC OXIDES**

CLASS 2 — **CLASS 1** — **CLASS 3**

(Higher sulphides) (Sulphides) (Acidic oxides) (Basic oxides) (Higher oxides)

Typical colour transition:

‘Dark’ Black (w. visible sulphide grains)/Black/Dark grey/Grey/Light Grey/White/Cream/Yellow/Light Brown/Brown/Dark Brown/Red

Increasing sulphide content ⟷ High silica content ⟷ Increasing oxide content

(Low silica content) (Low silica content)

Highly reducing (High R.P. /Low O.P.) ⟷ Neutral ⟷ Highly oxidizing (High O.P. /Low R.P.)

Oxidizing agent required — (Self-fluxing) — Reducing agent required

(Excess lead fusion/Soda ash/KNO$_3$) — (Standard flux w. Added flour + SiO$_2$ (acid flux))

R.P. = Reducing power

O.P. = Oxidizing power

Materials

Marking Pens

Sticky Labels

Samples provided by your supervisor

Full PPE including heavy duty acid resistant gloves

Spatula

Fumehood

Test tubes with test tube rack

Concentrated (15M) nitric acid (HNO_3) in auto dispenser

Waste bucket for spent samples and nitric acid

Method

1. Your lecturer (client) will provide you with a number of samples of different mineral origin.

   NOTE: Nitric acid will cause serious burns to the skin and eyes! Please read the MSDS and highlight its hazards and the proper PPE required and disposal instructions.

2. Place a small quantity of the samples (about 2g) into test tubes. You are not required to weigh the samples as this is a qualitative test, not a quantitative one.

3. Make a note as to the sample ID, colour, and expected sample class based on the colour of the sample. Place your observations in the table provided.

4. **NOTE: Prior to dispensing the nitric acid, get a labelled waste beaker and prime the auto dispenser by lifting the top of the dispenser and carefully push down to remove any air bubbles in the tubing.**

5. **Once the samples are in test tubes, pick up the rack and dispense about 1mL of the nitric acid whilst holding the rack, NOT the test tube!**

6. **Keep the test tube in the fume cupboard as some samples will react violently with the nitric acid.** Record your observations in the table provided.

7. Based on the results obtained, determine if the visual observation agrees with the nitric acid test to confirm the sample classification.

8. Clean up the test tubes by carefully rinsing any residues (sample and acid) into a labelled waste bucket.

9. Wash all equipment used carefully and place on the drying racks provided in the laboratory.

Results:

Test tube No.	Sample ID	Sample Colour	Sample class (1, 2 or 3) (observation)	Positive Nitric acid test? (Y/N)	Nitric acid test (Low, Medium, High)	Sample class (1, 2 or 3) (experiment)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						

Questions:

1. What is the name of the brown gas when nitric acid reacts with the samples?

2. Referring to the SDS what is the hazard class/classes for concentrated nitric acid?

3. What are the main hazards associated with this material?

4. What is the recommended PPE when using concentrated nitric acid?

5. What reagents are incompatible with nitric acid?

Elements of Competency		**Assessor's Comments**
Obtain the samples		
Prepare sample for testing		
Store backup samples		
Dispose of waste and spent samples		
Maintain a safe work environment		
I confirm that the above is my work and the assessor has given me sufficient feedback. **Candidate's signature:**		**Date:**
Candidate's comments:		
Assessor's signature:		**Date:**

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Assessment Task 1

Qualification national code and title	MSL40118 Certificate IV in Laboratory Techniques BDV3
Unit/s national code/s and title/s	MSL954005 Prepare Mineral Samples for Analysis

Assessment type (☑):

☒ Questioning (Oral/Written)

☒ Practical Demonstration

☐ 3rd Party Report

☐ Other – Project/Portfolio (*please specify)*

Assessment Resources:

- Relevant PPE (gloves, safety glasses, safety boots, lab coats)
- Prepare Mineral Samples training manual
- Sample preparation equipment
- Mineral samples for preparation

Assessment Instructions:

Assessment Title
Assessment 1 – Job Safety Analysis:
- Exercise 3 - Job Safety Analysis exercise

Assessment Description
This assessment requires you to perform a Job Safety Analysis prior to preparing your samples for analysis.

You are to inspect the sample preparation area and locate any potential hazards that are found within the preparation laboratory. This can involve pieces of equipment, samples being prepared, chemical reagents and any other hazards that can be associated with the preparation of the client samples provided.

Assessment Venue: C block

Assessment Time/duration: 3 weeks

Due Date: Session 7

Assessment Instrument:

SECTION B – JOB SAFETY ANALYSIS

ASSESSMENT 1

Exercise 2: JSA exercise

Introduction

- Definition(s): A **job safety analysis (JSA)** is a procedure which helps integrate accepted safety and health principles and practices into a particular task or job operation. In a JSA, each basic step of the job is to identify potential hazards and to recommend the safest way to do the job. Other terms used to describe this procedure are **job hazard analysis (JHA), hazardous task analysis (HTA)** and **job hazard breakdown**. (Wikipedia)

- In this exercise, a JSA will be conducted prior to performing any tasks within the sample preparation laboratory. This will involve inspecting all items of equipment, samples and the work surroundings to identify any potential hazards and risks that may exist within the preparation area.

- Use the table provided below to highlight the potential hazards found in the sample preparation laboratory prior to carrying out the work task/s of sample preparation. Take note of the sample types, equipment and surroundings and any potential risks associated with the samples being prepared and the risk controls that can be implemented to minimise any risks or exposures to yourself and others within the work area.

Elements of Competency

The following elements of competency are addressed in this exercise:

1 Interpret and schedule client requirements:

    1.4 Identify hazards, safety equipment and safe work procedures specified for the sample, preparation methods, reagents and equipment

Exercise 2: JOB SAFETY ANALYSIS – MINERAL PREPARATION LAB

JOB DESCRIPTION: PAGE: [1] OF [1] JSA NO: [1]			**DATE:**		☐ **NEW** ☐ **REVISED**
TITLE OF PERSON WHO UNDERTAKES JOB:		**SUPERVISOR:** Dominic Cooper		**PREPARATION BY:**	
CAMPUS/ DIVISION: East Perth Campus Science & Greenskills	**SECTION:** Mineral		**PLANT/LOCATION:** C block East Perth	**REVIEWED BY:** Dominic Cooper	
REQUIRED PERSONAL PROTECTIVE EQUIPMENT: **EMERGENCY SHUT-OFF SWITCHES LOCATION:**			**APPROVED BY SUPERVISOR?** ☐ Yes ☐ No **SUPERVISOR SIGNOFF:** **DATE:**		

STEP NO.	BASIC TASKS OF JOB	POTENTIAL HAZARDS	RISK CONTROL MEASURES RECOMMENDED	PERSON RESPSONSIBLE

STEP NO.	BASIC TASKS OF JOB	POTENTIAL HAZARDS	RISK CONTROL MEASURES RECOMMENDED	PERSON RESPSONSIBLE

STEP NO.	BASIC TASKS OF JOB	POTENTIAL HAZARDS	RISK CONTROL MEASURES RECOMMENDED	PERSON RESPSONSIBLE

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Assessment Task 1 Feedback

Assessment # and title	**Assessment 1 Job Safety Analysis**		
Lecturer name	Dominic Cooper		
Student name			
Student ID number			
Telephone contact number		**Email:**	

By completing and submitting this signed form to my lecturer, I am stating that:

a. The attached submission is completely my own work
b. I have correctly cited all sources of information used in this work (if required)
c. I have kept a copy of this assessment (where practicable)
d. I understand a copy of my assessment will be kept by the NMTAFE for their records
e. I understand my assessment may be selected for use in the NMTAFE's validation and audit process to ensure student assessment meets requirements

Student Signature		**Date**	

Assessor please note: Where verbal clarification has been sought from a student to gather additional assessment evidence from an assessment item, question/s and response/s must be recorded, signed, and dated by the assessor, against the relevant assessment item/s.

Task No.	Student is able to …	S or NYS	Comments
1	Wear appropriate PPE for the task being undertaken:		
a	• Dust coat		
b	• Hearing protection		
c	• Appropriate footwear		
d	• Gloves		
e	• Particle mask		
2	Identified hazards within the work environment on JSA		
3	Noted emergency shutoff switches near preparation equipment		
4	JSA satisfactorily completed and signed off		
Element No.	**Elements & Performance Criteria**		
1	**Interpret and schedule client requirements:**		
	1.4 Identify hazards, safety equipment and safe work procedures specified for the sample, preparation methods, reagents and equipment		

Submission 1 ☐ **Assessment Result Satisfactory / Not Yet Satisfactory** *(please circle)* **Date:** ___________

To satisfy requirements for this assessment, you need to complete the following:

Feedback to student:

Submission 2 ☐ **Assessment Result Satisfactory / Not Yet Satisfactory** *(please circle)* **Date:** ___________

To satisfy requirements for this assessment, you need to complete the following:

Feedback to student:

Student Feedback

Feedback from student:

Lecturer Signature: ____________ **Student Signature:** _________________________

Assessment Task 2

Qualification national code and title	MSL40118 Certificate IV in Laboratory Techniques BDV3
Unit/s national code/s and title/s	MSL954005 Prepare Mineral Samples for Analysis

Assessment type (☑):

☒ Questioning (Oral/Written)

☒ Practical Demonstration

☐ 3rd Party Report

☐ Other – Project/Portfolio (*please specify)*

Assessment Resources:

- Relevant PPE equipment (gloves, safety glasses, safety boots, lab coats)
- Prepare Mineral Samples training manual
- Sample preparation equipment

Assessment Instructions:

Assessment Title
Assessment 2 - Chain of Custody:

- Exercise 3 - Chain-of-Custody exercise

Assessment Description
This assessment requires you to select three different mineral sample types (ore, drill core and composite) that are located in the sample preparation lab.

A number of the relevant Chain of Custody forms for the samples provided are in the training manual. You are to compare the C-o-C forms in the training manual with those provided with the samples. If there are any errors, discrepancies or missing information between the two sets of forms, you are to highlight the differences in your training manual on the C-o-C forms provided.

NOTE: The correct C-o-C information can be found with the samples provided. The C-o-C forms for the Composite samples can be found in the "Chain of Custody forms – Composite samples" file on the top shelf where the Composite samples are located in the preparation laboratory.

Assessment Venue: C block

Assessment Time/duration: 3 weeks

Due Date: Session 7

Assessment Instrument:

SECTION C – CHAIN OF CUSTODY

ASSESSMENT 2

Exercise 3: Chain-of-Custody exercise

Introduction

- Definition(s): A process that tracks the movement of evidence through its collection, safeguarding, and analysis lifecycle by documenting each person who handled the evidence, the date/time it was collected or transferred, and the purpose for the transfer.(Wikipedia)

- In this exercise, samples will need a range of sample preparation method to prepare them for chemical analysis in the laboratory. For this to be performed, a chain-of-custody form needs to be referred to determine what type of preparation method and analytes are required as per the client's instructions.

The chain of custody forms provided in this manual closely reflect (not exactly, as they contain errors) the chain of custody forms in the samples provided in the sample preparation lab. You are to closely inspect and compare 3 chain of custody forms in the training manual with the same 3 samples provided to you and the chain of custody found with those samples. Any differences/errors found between the forms should be highlighted on the forms in your training manual.

Elements of Competency

The following elements of competency are addressed in this exercise:

1 Interpret and schedule client requirements

    1.1 Review client request to identify sample/analysis requirements, preparation methods and equipment involved

    1.2 Inspect samples, compare with specifications, record and report any discrepancies

    1.3 Liaise with client when samples and/or request forms do not comply with workplace procedures

Chain of Custody (COC) Form
NMTafe Laboratory
140 Royal Street, East Perth, phone 6211 6000

Client: Sandfire Resources/Microanalysis	**Site/Location:** Meekatharra	**Work to be undertaken:** Sample preparation and elemental analysis required	**Courier:** Toll
Lab Contact: Andrew James		**Sample preparation required** / **Elements to be determined**	**Phone Number:** 61-861411768
Address: Sandfire Resources, Meekatharra, WA, 6642			**Contact Name:** George Bennetts

Sample ID	NMTAFE ID	Sample Description	Matrix	Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Arsenic (As)	Lead (Pb)	**Preservation:** • Sealed container/ drums/plastic bag • Ambient temp. • Moisture free environment Comments
190432	202304001	Dark grey/black powder/grains	Copper concentrate (as copper sulphide)	X			X	X	X	X		X				**Caution:** High sulphide content. Care if heating! Dry at low temperature (~60 deg. C)

Special Instructions:
NOTE: Sample may be wet. May need to be dried before subsampling. Sample may stick to ring mill. Silica wash of mill may be required.

TAT:
Standard

Relinquished by: George Bennetts Signature: G.A.Bennetts	**Date:** 24/06/2019	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D.J.Cooper	**Date:** 24/06/2019	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: J202304-01	**Temperature:** 23 deg. C	**TAT required:** Same day/1/2/3/4/[✓ STD]	**Container Type:** Metal drum x Plastic drum Plastic bag Cloth bag Paper bag	**Security seal:** Intact/Broken/[✓ None]

Chain of Custody (COC) Form

NMTafe Laboratory

140 Royal Street, East Perth, phone 6211 6000

Client: Microanalysis	**Site/Location:** Esperance	**Work to be undertaken:** Sample preparation and elemental analysis required	**Courier:** Toll
Lab Contact: Andrew Jones			**Phone Number:** 61-861411768
Address: Alpha Gallant Natural State Reserves, Esperance, WA, 6450			**Contact Name:** George Bennetts

				Sample preparation required						Elements to be determined						
Sample ID	**NMTAFE ID**	**Sample Description**	**Matrix**	Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Arsenic (As)	Lead (Pb)	**Preservation:** - Sealed container/ drums/plastic bag - Ambient temp. - Moisture free environment **Comments**
190280 (ESP19_MT_004)	202304002	Red coarse powder/grains	Crude Iron oxide	X			X	X	X				X			**Caution:** High oxide content.

Special Instructions:
NOTE: Sample will stick to ring mill. Silica wash will be required!

TAT:
4 days

Relinquished by: George Bennetts Signature: G.A.Bennetts	**Date:** 28/05/2019	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D.J.Cooper	**Date:** 28/05/2019	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: J202304-02	**Temperature:** 23 deg. C	**TAT required:** Same day/1/2/3/[✓ 4]/STD	**Container Type:**	**Security seal:** Intact/Broken/[✓ None]

Container Type:	
Metal drum	
Plastic drum	x
Plastic bag	
Cloth bag	
Paper bag	

Chain of Custody (COC) Form
NMTafe Laboratory
140 Royal Street, East Perth, phone 6211 6000

Client: EMR Golden Grove/Microanalysis	**Site/Location:** Yalgoo	**Work to be undertaken:** Sample preparation and elemental analysis required	**Courier:** Toll
Lab Contact: R.Hughes/N.Pendragon			**Phone Number:** 61-899564283
Address: EMR Golden Grove, Yalgoo, Golden Grove, WA, 6635			**Contact Name:** George Bennetts

Preservation:
- Sealed container/ drums/plastic bag
- Ambient temp.
- Moisture free environment

Sample ID	NMTAFE ID	Sample Description	Matrix	Sample preparation required						Elements to be determined						Comments
				Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Arsenic (As)	Lead (Pb)	
190281 (NZ47939)	202304003	Grey/black powder	Zinc ore concentrate (as zinc sulphide)	X			X	X	X		X					**Caution:** High sulphide content. Care if heating! Dry at low temperature (~60 deg. C)

Special Instructions:
NOTE: Sample may be wet. May need to be dried before subsampling.

TAT:
3 days

Relinquished by: George Bennetts Signature: G.A.Bennetts	**Date:** 28/05/2019	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D.J.Cooper	**Date:** 28/05/2019	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: J202304-03	**Temperature:** 23 deg. C	**TAT required:** Same day/1/2/[✓ 3]/4/STD	**Container Type:**	**Security seal:** Intact/Broken/[✓ None]

Container Type:	
Metal drum	
Plastic drum	x
Plastic bag	
Cloth bag	
Paper bag	

Chain of Custody (COC) Form

NMTafe Laboratory

140 Royal Street, East Perth, phone 6211 6000

Client: Abra Mining/ ALS Metallurgy/Microanalysis	**Site/Location:** Gascoyne region (North Meekatharra)/East Perth	**Work to be undertaken:** Sample preparation and elemental analysis required	**Courier:** Toll
Lab Contact: Nimue Pendragon (Microanalysis)		**Sample preparation required** / **Elements to be determined**	**Phone Number:** +61 8 93442416
Address: ALS Metallurgy, 7 Macadam Place, Balcatta, WA, 6021			**Contact Name:** George Bennetts

Sample ID	NMTAFE ID	Sample Description	Matrix	Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Silver (Ag)	Arsenic (As)	Lead (Pb)	**Preservation:** - Sealed container/ drums/plastic bag - Ambient temp. - Moisture free Comments
190264 (ABRA Lead concentrate project, Gascoyne WA)	202304004	Red powder	Lead ore concentrate	X			X	X	X				X		X	**Caution:** Contains lead and high sulphide. Care if heating! Dry at low temperature (~60 deg. C)

Special Instructions:
NOTE: Sample may be wet. May need to be dried before subsampling. Wear appropriate PPE during preparation!

TAT:
1 day

Relinquished by: George Bennetts Signature: G.A.Bennetts	**Date:** 27/05/2019	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D.J.Cooper	**Date:** 27/05/2019	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: **J202304-04**	**Temperature:** 23 deg. C	**TAT required:** Same day/[✓ 1]/2/3/4/STD	**Container Type:** Metal drum: Plastic drum: x Plastic bag: Cloth bag: Paper bag:	**Security seal:** [✓ Intact]/Broken/None

Chain of Custody (COC) Form
NMTafe Laboratory
140 Royal Street, East Perth, phone 6211 6000

Client: BHP/Gemco	**Site/Location:** Groote Eylandt	**Work to be undertaken:** Sample preparation and elemental analysis required	**Courier:** Toll
Lab Contact:			**Phone Number:** +61 8 93442416
Address: Groote Eylandt Mining Company Pty. Ltd., Alyangula, Groote Eylandt NT, 0885			**Contact Name:** George Bennetts

Sample ID	NMTAFE ID	Sample Description	Matrix	Sample preparation required						Elements to be determined						**Preservation:** • Sealed container/ drums/plastic bag • Ambient temp. • Moisture free environment
				Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Manganese (Mn)	Lead (Pb)	Comments
CMT016	202304005	White/cream powder	Manganese ore concentrate aggregates	X	X		X	X	X				X	X		**Caution:** Contains high oxide.
				Special Instructions: NOTE: Sample may stick to ring mill. Silica wash may be required.												**TAT:** Standard

Relinquished by: George Bennetts Signature: G.A.Bennetts	**Date:** 27/05/2015	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D.J.Cooper	**Date:** 27/05/2015	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: **J202304-05**	**Temperature:** 23 deg. C	**TAT required:** Same day/1/2/3/4/[✓ STD]	**Container Type:**		**Security seal:** Intact/Broken/[✓ None]
			Metal drum		
			Plastic drum	x	
			Plastic bag		
			Cloth bag		
			Paper bag		

Chain of Custody (COC) Form
NMTafe Laboratory
140 Royal Street, East Perth, phone 6211 6000

Client: Dominion Mining Resources	**Site/Location:** Meekatharra	**Work to be undertaken:** Sample preparation and elemental analysis required	**Courier:** Toll
Lab Contact: c/- Dr. Frits Grader			**Phone Number:** +61 8 9426 6400
Address: Dominion Mining Ltd., 15 Outram St., West Perth, WA, 6005			**Contact Name:** Dr. Frits Grader

Preservation:

- Sealed container/ drums/plastic bag
- Ambient temp.
- Moisture free environment

Sample ID	NMTAFE ID	Sample Description	Matrix	Sample preparation required						Elements to be determined						Comments
				Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Manganese (Mn)	Lead (Pb)	
31355 (Mickey Doolan #21)	202304006	Light grey dusty fine powder	Gold ore sulphide			X	X	X	X			X	X			**Caution:** Contains high sulphide and odour of xanthate. Adequate ventilation required!

Special Instructions:
NOTE: Very dusty sample. Wear appropriate PPE and supply adequate airflow and extraction fans during preparation steps.

TAT:
Same day

Relinquished by: Dr. Frits Grader Signature: Dr. F. Grader	**Date:** 07/08/1995	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D.J. Cooper	**Date:** 30/04/2015	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: **J202304-06**	**Temperature:** 23 deg. C	**TAT required:** [✓ Same day]/1/2/3/4/STD	**Container Type:**	**Security seal:** Intact/Broken/[✓ None]

Container Type:	
Metal drum	
Plastic drum	x
Plastic bag	
Cloth bag	
Paper bag	

Chain of Custody (COC) Form
NMTafe Laboratory
140 Royal Street, East Perth, phone 6211 6000

Client: NMTAFE	Site/Location: Various	Work to be undertaken: Sample preparation and elemental analysis required	Courier: In-house

Lab Contact:	Sample preparation required	Elements to be determined	Phone Number: +61 8 6211 6000
Address: 140 Royal Street, East Perth, WA, 6004, phone 6211 6000			Contact Name: Noeline Ellis

Sample ID	NMTAFE ID	Sample Description	Matrix	Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Manganese (Mn)	Lead (Pb)	Comments
Composite #13 (2020/21)	202304007	Dark grey dusty fine powder	Sulphide composite blend			X	X	X	X?	X	X	X	X		X	**Caution:** Contains high sulphides Adequate ventilation required!

Preservation:
- Sealed container/ drums/plastic bag
- Ambient temp.
- Moisture free environment

Special Instructions:
NOTE: Roll mix for 30 minutes prior to rotary splitting. Sample may require pulverizing!

TAT:
Standard

Relinquished by: Noeline Ellis Signature: N. Ellis	Date: 1/01/2020	Time: 9.00am	Received by: Dominic Cooper NMTafe Assay Dept. Signature: D. J. Cooper	Date: 1/01/2021	Time: 4.30pm	Received By (Company): NMTAFE Print name: Date & Time: Sign:	Supervisors signature: Date: Time:

Lab use only

Job number: **J202304-07**	Temperature: 23 deg. C	TAT required: Same day/1/2/3/4/[✓ STD]	Container Type:	Security seal: Intact/Broken/[✓ None]

Container Type:	
Metal drum	
Plastic drum	x
Plastic bag	
Cloth bag	
Paper bag	

Chain of Custody (COC) Form
NMTafe Laboratory
140 Royal Street, East Perth, phone 6211 6000

Client: NMTAFE	**Site/Location:** Various	**Work to be undertaken:** Sample preparation and elemental analysis required	**Courier:** In-house
Lab Contact: Noeline Ellis			**Phone Number:** +61 8 6211 6000
Address: 140 Royal Street, East Perth, WA, 6004, phone 6211 6000			**Contact Name:** Noeline Ellis

				Sample preparation required						Elements to be determined						
Sample ID	**NMTAFE ID**	**Sample Description**	**Matrix**	Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Manganese (Mn)	Lead (Pb)	**Comments**
Composite #14 (2020/22)	202304008	Dark grey dusty fine powder	Sulphide composite blend			X	X	X	X?	X	X	X	X		X	**Caution:** Contains high sulphides Adequate ventilation required!

Preservation:
- Sealed container/ drums/plastic bag
- Ambient temp.
- Moisture free environment

Special Instructions:
NOTE: Roll mix for 30 minutes prior to rotary splitting. Sample may require pulverizing!

TAT:
Standard

Relinquished by: Noeline Ellis Signature:	**Date:** 1/01/2020	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D.J.Cooper	**Date:** 1/01/2022	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: **J202304-08**	**Temperature:** 23 deg. C	**TAT required:** Same day/1/2/3/4/[✓ STD]	**Container Type:**	**Security seal:** [✓ Intact]/Broken/None

Container Type:

Metal drum	
Plastic drum	x
Plastic bag	
Cloth bag	
Paper bag	

Chain of Custody (COC) Form
NMTafe Laboratory
140 Royal Street, East Perth, phone 6211 6000

Client: NMTAFE	**Site/Location:** Various	**Work to be undertaken:** Sample preparation and elemental analysis required	**Courier:** In-house
Lab Contact: Lyall Offe			**Phone Number:** +61 8 6211 6000
Address: 140 Royal Street, East Perth, WA, 6004, phone 6211 6000			**Contact Name:** Lyall Offe

				Sample preparation required						Elements to be determined						
Sample ID	NMTAFE ID	Sample Description	Matrix	Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Manganese (Mn)	Lead (Pb)	Comments
Drill Core	202304009	Grey/white drill core samples	Granitic core samples – may contain gold		X	X		X	X	X	X	X	X			**Caution:** Possible sharp ends on drill core.

Preservation:
- Sealed container/ drums/plastic bag
- Ambient temp.
- Moisture free environment

Special Instructions:
NOTE: Use of jaw crusher required! Hearing, glove and eye protection required!

TAT:
Standard

Relinquished by: Lyall Offe Signature: L.Offe	**Date:** 1/01/2020	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D.J.Cooper	**Date:** 1/01/2020	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: **J202304-09**	**Temperature:** 23 deg. C	**TAT required:** Same day/1/2/3/4/[✓ STD]	**Container Type:**	**Security seal:** Intact/Broken/[✓ None]

Container Type:	
Metal drum	
Plastic drum	x
Plastic bag	
Cloth bag	
Paper bag	

Chain of Custody (COC) Form
NMTafe Laboratory
140 Royal Street, East Perth, phone 6211 6000

Client: Dominion Mining Resources	**Site/Location:** Meekatharra	**Work to be undertaken:** Sample preparation and elemental analysis required	**Courier:** Toll
Lab Contact: c/- Dr. Frits Grader			**Phone Number:** +61 8 9426 6400
Address: Dominion Mining Ltd., 15 Outram St., West Perth, WA, 6005			**Contact Name:** Dr. Frits Grader

Sample ID	NMTAFE ID	Sample Description	Matrix	Sample preparation required						Elements to be determined						**Preservation:** • Sealed container/ drums/plastic bag • Ambient temp. • Moisture free environment
				Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Nickel (Ni)	Lead (Pb)	Comments
37823 (Prohibition #20)	202304010	Grey black fine powder	Nickel sulphide (containing gold ore)	X			X	X	X			X	X	X		**Caution:** Contains high sulphide and nickel. Adequate ventilation required!

Special Instructions:
NOTE: Sample contains nickel. Wear appropriate PPE and supply adequate airflow and extraction fans during preparation steps.

TAT:
2 days

Relinquished by: Dr. Frits Grader Signature: Dr. F. Grader	**Date:** 07/08/1995	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D. J. Cooper	**Date:** 30/04/2015	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: **J202304-10**	**Temperature:** 23 deg. C	**TAT required:** Same day/1/[✓ 2]/3/4/STD	**Container Type:**	**Security seal:** Intact/Broken/[✓ None]

Container Type:	
Metal drum	
Plastic drum	x
Plastic bag	
Cloth bag	
Paper bag	

Chain of Custody (COC) Form
NMTafe Laboratory
140 Royal Street, East Perth, phone 6211 6000

Client: NMTAFE	**Site/Location:** Various	**Work to be undertaken:** Sample preparation and elemental analysis required		**Courier:** In-house
Lab Contact: Noeline Ellis		**Sample preparation required**	**Elements to be determined**	**Phone Number:** +61 8 6211 6000
Address: 140 Royal Street, East Perth, WA, 6004, phone 6211 6000				**Contact Name:** Noeline Ellis

Sample ID	NMTAFE ID	Sample Description	Matrix	Floor mixing	Jaw crushing	Roll mixing	Rotary splitting	Riffle splitting	Pulverizing	Copper (Cu)	Zinc (Zn)	Gold (Au)	Iron (Fe)	Manganese (Mn)	Lead (Pb)	**Preservation:** • Sealed container/ drums/plastic bag • Ambient temp. • Moisture free environment Comments
Composite #11 (Sept.2019)	202304011	Dark grey dusty fine powder	Sulphide composite blend		X	X	X	X	X?	X	X	X	X		X	**Caution:** Contains high sulphides Adequate ventilation required!

Special Instructions:
NOTE: Roll mix for 30 minutes prior to rotary splitting. Sample may require pulverizing!

TAT:
Standard

Relinquished by: Noeline Ellis Signature:	**Date:** 1/01/2020	**Time:** 9.00am	**Received by:** Dominic Cooper NMTafe Assay Dept. Signature: D.J.Cooper	**Date:** 1/01/2022	**Time:** 4.30pm	**Received By (Company):** NMTAFE **Print name:** **Date & Time:** **Sign:**	**Supervisors signature:** **Date:** **Time:**

Lab use only

Job number: **J202304-08**	**Temperature:** 23 deg. C	**TAT required:** Same day/1/2/3/4/STD	**Security seal:** Intact/Broken/None

Metal drum	
Plastic drum	x
Plastic bag	
Cloth bag	
Paper bag	

Government of Western Australia
North Metropolitan TAFE

Assessment Task 2 Feedback

Assessment # and title	**Assessment 1 Job Safety Analysis**		
Lecturer name	Dominic Cooper		
Student name			
Student ID number			
Telephone contact number		**Email:**	

By completing and submitting this signed form to my lecturer, I am stating that:

a. The attached submission is completely my own work
b. I have correctly cited all sources of information used in this work (if required)
c. I have kept a copy of this assessment (where practicable)
d. I understand a copy of my assessment will be kept by the NMTAFE for their records
e. I understand my assessment may be selected for use in the NMTAFE's validation and audit process to ensure student assessment meets requirements

Student Signature		**Date**	

Assessor please note: Where verbal clarification has been sought from a student to gather additional assessment evidence from an assessment item, question/s and response/s must be recorded, signed, and dated by the assessor, against the relevant assessment item/s.

Task No.	Student is able to …	S or NYS	Comments
1	Completed COC form for ore sample and interpreted requirements for preparation		
2	Completed COC form for composite sample and interpreted requirements for preparation		
3	Completed COC form for drill core and interpreted requirements for preparation		
4	Highlight/record on the COC forms (in the training manual) any discrepancies on the sample containers		
5	Note special safety requirements for the samples being prepared		
Element No.	**Elements & Performance Criteria**		
1	**Interpret and schedule client requirements:**		
	1.1 Review client request to identify sample/analysis requirements, preparation methods and equipment involved		
	1.2 Inspect samples, compare with specifications, record and report any discrepancies		
	1.3 Liaise with client when samples and/or request forms do not comply with workplace procedures		

Submission 1 ☐ **Assessment Result Satisfactory / Not Yet Satisfactory** *(please circle)* **Date:** ______

To satisfy requirements for this assessment, you need to complete the following:

Feedback to student:

Submission 2 ☐ **Assessment Result Satisfactory / Not Yet Satisfactory** *(please circle)* **Date:**

____

To satisfy requirements for this assessment, you need to complete the following:

Feedback to student:

Student Feedback

Feedback from student:

Lecturer Signature: ____________________ **Student Signature:** ___________________

Assessment Task 3

Qualification national code and title	MSL40118 Certificate IV in Laboratory Techniques BDV3
Unit/s national code/s and title/s	MSL954005 Prepare Mineral Samples for Analysis

Assessment type (☑):

☒ Questioning (Oral/Written)

☒ Practical Demonstration

☐ 3rd Party Report

☐ Other – Project/Portfolio (*please specify)*

Assessment Resources:

- Relevant PPE equipment (gloves, safety glasses, safety boots, lab coats)
- Prepare Mineral Samples training manual
- Sample preparation equipment

Assessment Instructions:

Assessment Title
Assessment 3 - Mineral Dressing & Sample Preparation:
- Exercise 4 – Jaw Crushing, Mixing, Splitting and Pulverising Techniques

Assessment Description
This assessment requires you to select three different mineral sample types (ore, drill core and composite) that are located in the sample preparation lab.

A number of the relevant Chain of Custody forms for the samples provided are in the training manual. You are to compare the C-o-C forms in the training manual with those provided with the samples. If there are any errors, discrepancies or missing information between the two sets of forms, you are to highlight the differences in your training manual on the C-o-C forms provided.

NOTE: The correct C-o-C information can be found with the samples provided. The C-o-C forms for the Composite samples can be found in the "Chain of Custody forms – Composite samples" file on the top shelf where the Composite samples are located in the preparation laboratory.

Assessment Venue: C block

Assessment Time/duration: 3 weeks

Due Date: Session 7

SECTION D - MINERAL DRESSING & SAMPLE PREPARATION

ASSESSMENT 3

Exercise 4: Jaw Crushing, Mixing, Splitting and Pulverising Techniques

Introduction

To obtain reproducible results it is essential that laboratories use standardised procedures for the preparation of samples. These procedures will not necessarily be the same for each sample but may comprise various combinations of the following treatments to obtain a representative sample.

- Separation and removal of extraneous components (segregation)
- Drying
- Jaw Crushing
- Homogenising
- Grinding
- Sieving and
- Reducing sample size by splitting

In this practical exercise, samples will be homogenised and split using a range of techniques depending on the sample size and type of sample submitted by the client. Refer to the sample C-o-C form to determine what methods are required.

The techniques required for homogenisation include floor mixing with a shovel or a bottle roller. For samples provided in excess of 2kg, a rotary splitter can be used to reduce the sample mass in conjunction with riffling techniques to produce homogeneous **duplicate** portions of around 100g (± ~20g) for analysis.

Elements of Competency

The following elements of competency are addressed in this exercise:

- Interpret and schedule client requirements
- Prepare client samples for analysis
- Use non-destructive methods to prepare laboratory portions for analysis
- Maintain a safe work environment

Materials

Marking Pens

Sticky Labels

Samples provided by your supervisor

PPE

Jaw crusher

Rotary splitter

Riffle box

Sample bags

Top loading balance

Shovels

Rubber mixing mats

Note:

Please wear the following PPE for this exercise:

Safety boots

Dust coat

Safety glasses

Dust mask

Hearing protection

Disposable gloves

Method – Introduction

1. Your lecturer (client) will provide you with a number of samples of different mineral type.

   Sample 1 – Ore sample

   Sample 2 – Drill core sample

   Sample 3 – Composite sample

   Sample 4 – Silica sand samples (used as a 'wash' between samples)

2. This practical involves the use of a jaw crusher, a large rotary splitting device, a bottle roller and a pulveriser. Your lecturer will explain their operation and the safety features prior to their use. **No student is to use the devices prior to receiving instruction from the supervisor.** Both the rotary and riffle splitters as well as the jaw crusher must be clean prior to their use. This involves the use of a high-pressure air gun to remove traces of dust that may be present on the equipment.

3. **NOTE: Sample masses can exceed 20kg! Proper lifting techniques need to be used. Remember to wear appropriate footwear, lab coats, dust mask, gloves, safety glasses and hearing protection.**

4. If large samples are provided, the sample preparation area (i.e. laboratory floor) must be cleaned prior to sample treatment. Use the industrial vacuum cleaner provided to minimise the generation of airborne dusts and particulate matter.

5. Take note of the sample type, client name, sample identification number/s, NMTAFE ID and weight of container using the balances provided. Record the information in the table below.

Sample type	Client name	Sample ID	NMTAFE ID	Container Weight (g or kg)
Ore sample + container				
Composite sample + container				
Drill core				

Method – Part A – Rotary mixing – Ore sample

1. Tip the contents of the ore sample bucket onto the floor and homogenise by mixing thoroughly using a shovel. Any large particles that may have agglomerated together can be broken down with a hammer or similar object to ensure good homogenization of the sample.

2. Transfer the contents back into the bucket and tip the contents into the rotary splitter opening (hopper) that has been previously cleaned using an air gun.

3. Turn the rotary splitter on to segregate the sample into eight approximately equal portions. Once the sample has completely split, select one of the portions and record the weight in the rotary split results table (1st pass) below. If the mass of the sample exceeds 1kg, the sample will need to be placed into the rotary splitter again to reduce the sample size down to a more manageable level. If the sample needs rotary splitting again, place the contents of the portion back into the rotary opening (hopper) and split again.

4. Weigh the rotary split portion again (2nd pass) and record the mass in the table provided. If the mass exceeds 1kg of sample, repeat for a 3rd pass if required.

5. Return the seven remaining rotary split samples back into the original sample bucket.

6. Place the rotary split portion into a riffle splitter where the sample will pass into two separate collection containers (‘A’ and ‘B’). Weigh the ‘A’ sample fraction and record your data in the training manual.

7. Continue the riffle splitting process until two final ‘A’ fractions of around approximately 100g is obtained, recording the sample weights each time. These can be labelled as ‘A1’ and ‘A2’ in paper sample packets provided. Be sure to include sample details, date, group name and analytes required.

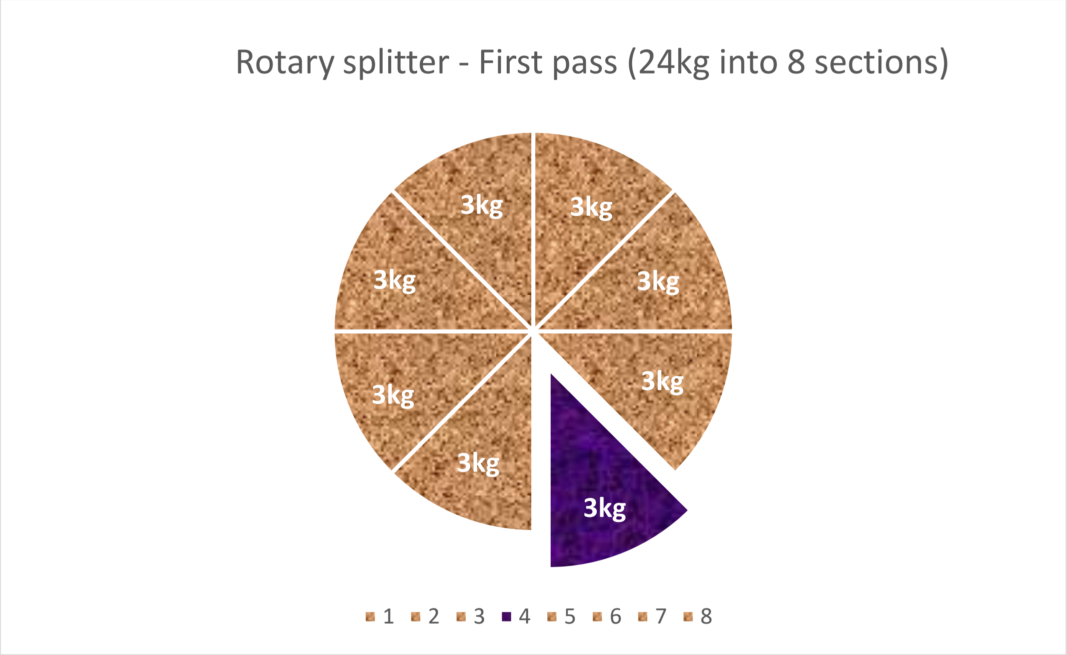
8. The two samples are now ready for pulverising. See Part D below.

Rotary	Weight of Section (g)	Weight of Section + sample (g)	Weight of sample (g)
First pass			
Second pass			
Third pass (if required)			

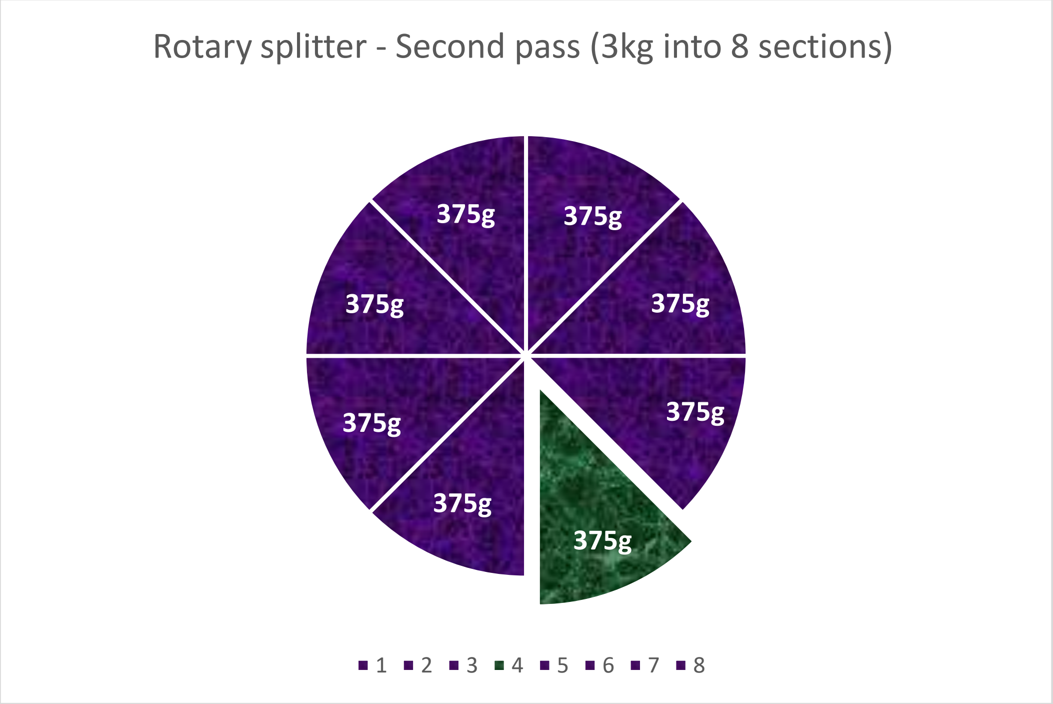
Weights	**Split 1** **First pass**	**Split 2** **Second pass**	**Split 3** **Third pass (if required)**
Weight of Section A (kg) (empty)			
Weight of Section A + sample (kg)			
Weight of sample (g)			
Duplicate			
Weight of Section B (kg) (empty)		**Not required**	**Not required**
Weight of Section B + sample (kg)		**Not required**	**Not required**
Weight of sample (g)		**Not required**	**Not required**

Visual example - Rotary splitting and riffle splitting sequence diagram for a 24kg sample:

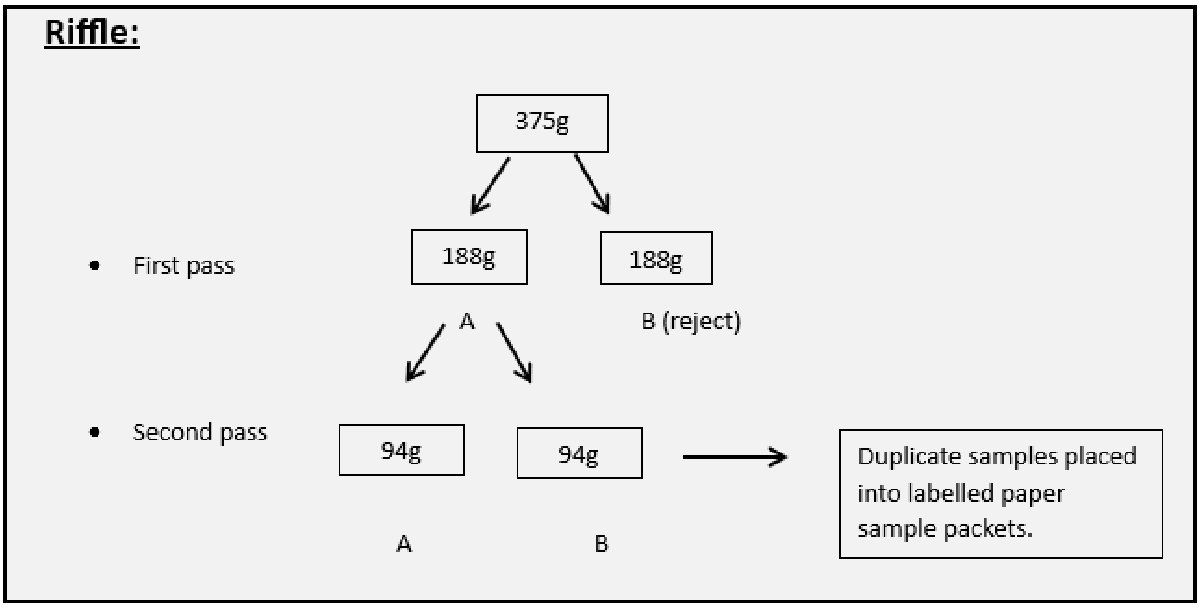
1.



2.



3.



Duplicate samples placed into labelled paper sample packets.

Method – Part B – Jaw crushing - Drill core sample

Sample Name	
TAFE ID	
Weight sample	
Weight of 'Drill core A' after splitting	
Weight of 'Drill core B' after splitting	

1. You are provided with either a drill core or rock sample.
2. **Ensure a JSA has been completed and signed off by your supervisor prior to using this equipment!**
3. Record the sample details and the weight of the sample in the table above.
4. Your lecturer will demonstrate the safe operation of the jaw crushing device.
5. Ensure the crusher has been cleaned using an air gun to remove any particulate matter from previous samples to minimise contamination.
6. Turn crusher on, remembering to keep hands clear of the equipment at all times. NOTE: Wear hearing protection, safety glasses, dust mask and gloves
7. Place the sample into the crusher and cover the opening with a metal plate to prevent sample escaping out of the crusher during the crushing process, which should take no more than 15 seconds, depending on the sample size.
8. Once crushed, collect the sample from the collection bucket and transfer the contents into a metal tray.
9. Split the sample into two portions labelled 'Drill core A' and 'Drill core B' using a riffle splitter and place the two portions into two separate sample packets. Weigh the two portions after splitting and record the values in the table above.
10. The crushed samples are now ready for pulverising using a ring mill pulveriser, as per Part D below.
11. Wash hands thoroughly.

Method – Part C – Bottle roll mixer – Composite sample

1. You are provided with one of three composite samples.

2. **Ensure a JSA has been completed and signed off by your supervisor prior to using this equipment!**

3. Record the sample details into the table provided below.

Bottle roll mixer – Composite sample		**Weights (kg or g)**
Sample Name		
TAFE ID		
Container		
Weight sample + plastic container		
Weight of plastic container (empty)		
Weight of sample		
Rotary splitter		
First pass	**Weight of sample in rotary + 1 section**	
	Weight of section (empty)	
	Weight of sample	
Second pass	**Weight of sample in rotary + 1 section**	
	Weight of section (empty)	
	Weight of sample	
Riffle splitter		
A fraction	**Weight of 'A fraction' (empty)**	
	Weight of 'A fraction' + sample	
	Weight of Composite sample A	
B fraction	**Weight of 'B fraction' (empty)**	
	Weight of 'B fraction' + sample	
	Weight of Composite sample B	
C fraction **(if required)**	**Weight of 'A fraction' (empty)**	
	Weight of 'A fraction' + sample	
	Weight of Composite sample C	

4. Place the sample/s on the roll mixer and mix for 30 minutes to ensure homogenisation.

5. Remove the sample container and place the contents slowly through the hopper on the rotary splitter to reduce the sample size.

6. Turn the rotary splitter on to segregate the sample into eight approximately equal portions. Once the sample has completely split, select one of the portions and record in the table above. If the mass of the sample exceeds 1kg, the sample will need to be placed into the rotary splitter again to reduce the sample to a more manageable size.

7. Weigh the rotary split sample and place into a riffle splitter where the sample will pass into two separate collection containers ('A' and 'B'). Weigh the 'A' portion and record the weights in the table provided above. (There is no need to record the 'B' fraction weight, as this sample will be returned to the original sample bucket).

8. Continue to split the 'A' portion sample until 2 portions of around 100g is achieved. Record the weights in the table provided.

9. Return the 'B' portion and the seven remaining rotary split samples and any unused or excess portions of sample into the original client bucket.

10. Label the two samples as 'Comp1' and 'Comp2' on the paper sample packets provided. Include sample details, date, group name and analytes required.

11. The two samples are now ready for pulverising, **if required**. Check the samples for fineness by rubbing a small portion between your thumb and forefinger. See Part D below.

12. Wash hands thoroughly.

Method – Part D – Pulverising of samples & silica wash samples

1. Your supervisor will now demonstrate how to operate the pulveriser safely and the duration required to satisfactorily prepare the samples in readiness for the next step of the sample treatment process.

2. **Ensure a JSA has been completed and signed off by your supervisor prior to using this equipment!**

3. Transfer the samples into a ring mill and pulverise the samples for around 20-30 seconds to give a particle size of approximately 75µM. To check this, take some sample between the thumb and forefinger to feel the sample's consistency after pulverising, which should be like a fine powder, similar to talc or baby powder.

4. If the sample contains any coarse grains or still feels 'gritty', further pulverising time will be required, possibly another 15 to 20 seconds. CAUTION: Avoid over-pulverising the sample, as this will cause the sample to agglomerate and stick excessively to the ring mill making it heterogeneous and 'unfit for purpose'.

5. Once pulverising is completed, transfer the samples (A & B) back into their respective labelled paper packets.

6. Record the weights of all the prepared samples in their sample packets and place in the table below.

7. Place the samples in the provided yellow plastic sample bag for submission to the laboratory for analysis.

8. Clean the ring mill by doing a 'silica wash' by adding a scoop of silica sand into the ring mill and pulverising for approximately 30 seconds. Remove the silica from the bowl and collect into a paper packet labelled 'silica wash'. Place the silica wash sample into yellow plastic sample bag for future analysis.

9. Prior to leaving the laboratory, vacuum the entire work area with the provided vacuum cleaner.

10. Store all equipment in the designated areas and dispose of gloves, dust masks, ear plugs in the bin provided.

11. Wash hands thoroughly before leaving the sample preparation area.

Sample type	**Client name**	**Sample ID**	**NMTAFE ID**	**Final Sample packet weights (g)**
Ore sample A				
Ore sample B				
Ore sample Silica wash				
Composite sample A				
Composite sample B				
Composite sample silica wash				
Drill core A				
Drill core A				
Drill core silica wash				

Sampling Equipment – sketch a diagram of the equipment/process used to prepare the samples:
Rotary splitter
Riffle Splitter

Jaw crusher

Bottle roll mixing

Questions:

1.a) List at least five hazards in the use of the rotary splitter. What precautions are required when using this piece of machinery?

____

____

____

____

____

____

1.b) List three hazards associated with the use of a jaw crusher. What precautions are required when using this piece of machinery?

____

____

____

____

2. Define the following terms with regard to sampling:

a) Homogeneous

____

____

____

____

____

b) Heterogeneous

____

____

____

____

____

____

c) Integrity

____

____

____

____

____

____

d) Segregation

____

____

____

____

____

____

Post-Lab Exercise

1. Record any preparation difficulties that you may have encountered during the exercise. What precautions were taken to avoid contamination of the sample?

____

____

____

____

2. If contamination was to occur during testing or sample preparation, what action must be taken?

____

____

____

3. Describe the appropriate OHS procedures that were followed during this exercise.

4. Was there any departure from the preparation methods used or client specifications?

Elements of Competency		**Assessor’s Comments**
Obtain the samples		
Prepare sample for testing		
Store backup samples		
Dispose of waste and spent samples		
Maintain a safe work environment		
I confirm that the above is my work and the assessor has given me sufficient feedback. **Candidate’s signature:**		**Date:**
Candidate’s comments:		
Assessor’s signature:		**Date:**

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Assessment Task 3 Feedback

Assessment # and title	**Assessment 3 – Jaw Crushing, Mixing, Splitting and Pulverising Techniques**		
Lecturer name	Dominic Cooper		
Student name			
Student ID number			
Telephone contact number		**Email:**	

By completing and submitting this signed form to my lecturer, I am stating that:

a. The attached submission is completely my own work
b. I have correctly cited all sources of information used in this work (if required)
c. I have kept a copy of this assessment (where practicable)
d. I understand a copy of my assessment will be kept by the NMTAFE for their records
e. I understand my assessment may be selected for use in the NMTAFE's validation and audit process to ensure student assessment meets requirements

Student Signature		**Date**	

Assessor please note: Where verbal clarification has been sought from a student to gather additional assessment evidence from an assessment item, question/s and response/s must be recorded, signed, and dated by the assessor, against the relevant assessment item/s.

Task No.	Student is able to …	S or NYS	Comments
1	Wear appropriate PPE for the task being undertaken		
2	Engineering controls (extraction system, dust hoods) in operation to minimize dust exposure and wastes		
3	Plan & assemble required equipment and check if 'fit-for-purpose' for preparation of at least 3 different ore samples, to be prepared in parallel for optimisation of workflow		
4	Homogenise sample by floor mixing		
5	Homogenise sample by using bottle roller		
6	Reduce sample particle size by using a jaw crusher		
7	Reduce sample size by using a rotary splitter		
8	Reduce sample size by using a riffle splitter		
9	Reduce particle size by use of a ring mill pulveriser & check sample consistency/particle size with thumb and forefinger and adjust, if necessary, pulverisation times accordingly		
10	Monitor any build up of residues and risk of contamination on equipment and perform a silica wash between samples		

11	Note any departure from preparation methods and preparation difficulties or invalid steps to be to be highlighted on C-o-C form		
12	Prepare, label & bag and store at least 3 sample types using non-destructive techniques and present them for analysis with updated CoC information provided		
13	Review each method and preparation step and identify critical steps that can affect quality of results, safety and contamination		
14	Clean-up & replace equipment in work area and dispose of wastes responsibly		
15	Follow safety guidelines to prepare the samples safely as per supervisor's instructions/SOPs, ensuring good precision by maintaining sample integrity and a safe work environment in the process		
Element No.	**Elements & Performance Criteria**		
1	**Interpret and schedule client requirements:**		
	1.5 Plan parallel work sequences to optimise throughput of multiple sets of samples		
	1.6 Assemble all required equipment materials, reagents and check they are fit for purpose		
2	**Prepare client samples for analysis**		
3	**Use non-destructive methods to prepare laboratory portions for analysis**		
4	**Maintain a safe work environment**		

Submission 1 ☐ **Assessment Result Satisfactory / Not Yet Satisfactory** *(please circle)* **Date:** ___________

To satisfy requirements for this assessment, you need to complete the following:

Feedback to student:

Submission 2 ☐ **Assessment Result Satisfactory / Not Yet Satisfactory** *(please circle)* **Date:** ___________

To satisfy requirements for this assessment, you need to complete the following:

Feedback to student:

Student Feedback

Feedback from student:

Lecturer Signature: ____________________ **Student Signature:** ___________________

SECTION E – SAMPLE SUBMISSION

Exercise 5a: Sample Submission – Client 1

SAMPLE ANALYSIS SUBMISSION FORM

Client Information

Company Name	
Contact Name	
Address	
City, State, Postcode	
Phone Number	

Submit to (Tick the appropriate Boxes):

☐ Weighing Room

☐ Aqua Regia (Fe, As, Mn, Pb, Zn, Ni, Cu – Method AN140A)

☐ Aqua Regia/DIBK (Au – Method AN240A)

☐ Pre-roast/Aqua Regia/DIBK (Au – Method AN241A)

☐ Fusion Room (Fe, Mn, Pb, Zn, Ni, Cu – Method AN200A)

☐ Multi-acid digest (Multi elements - Fe, As, Mn, Pb, Zn, Ni, Cu – Method AN115A)

☐ XRD/XRF (Fe, Ca, Mg, Mn, Pb, Ni, Cu)

☐ Fire Assay (Au – Method AN300A)

☐

Three acid digest (Multi elements - Fe, As, Mn, Pb, Zn, Ni, Cu – Method AN120A)

Job Number: **Date:**

Sample ID #	Description	NMTAFE Sample ID#	Analyte(s) (Elements required)	OHS Notes/Hazards
(Silica wash)				

Exercise 5b: Sample Submission – Client 2

SAMPLE ANALYSIS SUBMISSION FORM

Client Information

Company Name	
Contact Name	
Address	
City, State, Postcode	
Phone Number	

Submit to (Tick the appropriate Boxes):

☐ Weighing Room

☐ Aqua Regia (Fe, As, Mn, Pb, Zn, Ni, Cu – Method AN140A)

☐ Aqua Regia/DIBK (Au – Method AN240A)

☐ Pre-roast/Aqua Regia/DIBK (Au – Method AN241A)

☐ Fusion Room (Fe, Mn, Pb, Zn, Ni, Cu – Method AN200A)

☐ Multi-acid digest (Multi elements - Fe, As, Mn, Pb, Zn, Ni, Cu – Method AN115A)

☐ XRD/XRF (Fe, Ca, Mg, Mn, Pb, Ni, Cu)

☐ Fire Assay (Au – Method AN300A)

☐ Three acid digest (Multi elements - Fe, As, Mn, Pb, Zn, Ni, Cu – Method AN120A)

Job Number: **Date:**

Sample ID #	Description	NMTAFE Sample ID#	Analyte(s) (Elements required)	OHS Notes/Hazards
(Silica wash)				

Exercise 5c: Sample Submission – Client 3

SAMPLE ANALYSIS SUBMISSION FORM

Client Information

Company Name	
Contact Name	
Address	
City, State, Postcode	
Phone Number	

Submit to (Tick the appropriate Boxes):

☐ Weighing Room

☐ Aqua Regia (Fe, As, Mn, Pb, Zn, Ni, Cu – Method AN140A)

☐ Aqua Regia/DIBK (Au – Method AN240A)

☐ Pre-roast/Aqua Regia/DIBK (Au – Method AN241A)

☐ Fusion Room (Fe, Mn, Pb, Zn, Ni, Cu – Method AN200A)

☐ Multi-acid digest (Multi elements - Fe, As, Mn, Pb, Zn, Ni, Cu – Method AN115A)

☐ XRD/XRF (Fe, Ca, Mg, Mn, Pb, Ni, Cu)

☐ Fire Assay (Au – Method AN300A)

☐ Three acid digest (Multi elements - Fe, As, Mn, Pb, Zn, Ni, Cu – Method AN120A)

Job Number: **Date:**

Sample ID #	Description	NMTAFE Sample ID#	Analyte(s) (Elements required)	OHS Notes/Hazards
(Silica wash)				

Questions:

5. Explain the importance of a sample submission form. What is its main purpose in a laboratory environment?

Elements of Competency		**Assessor's Comments**
Obtain the samples		
Prepare sample for testing		
Store backup samples		
Dispose of waste and spent samples		

Maintain a safe work environment		
I confirm that the above is my work and the assessor has given me sufficient feedback. **Candidate’s signature:**		**Date:**
Candidate’s comments:		
Assessor’s signature:		**Date:**

SECTION F - MINERAL DIGESTION

Exercise 6: Method AN140A - Aqua Regia Digestion for Base Metals

Introduction

Once samples have been sorted, crushed, split and pulverised, they are ready for the next part in the analytical process. Sample treatment can involve a number of processes depending on the mineral type being analysed and/or the analyte being sought in the sample. In this exercise, samples previously prepared will be treated in a number of different ways depending on the analyte being determined. Note the client job sheets for analytes requested and the preparation/pre-treatment methods required.

Aim

To determine the amount of base metals found in mineral samples, using aqua regia digestion.

Background

In geochemical analysis, some clients request a range of elements to give them an idea as to other metals that may have some commercial value. A simple yet effective method is to digest some of the mineral sample with aqua regia. This will leach the easily solubilised analytes (chemical elements) found in the sample and provide the client with a ‘ball park’ figure as to the levels of these analytes. The more resistant minerals such as refractory's (e.g. silica, magnetite, chromite and rare earths) will either not be attacked by the acid mixture or only be partially digested. In this exercise, a base metal analysis will be performed on the mineral samples for the analyte iron using aqua regia. The results obtained in this practical can be compared to those obtained in ‘Sample Treatment 3 – AN200A – the Sodium Peroxide method’.

Safety

Caution: Concentrated Nitric and Hydrochloric acids. Read MSDS before commencing the practical work. Wear gloves, lab coat and safety glasses and carry out digestion procedures in fume hoods provided. Ensure all PPE is ‘fit for purpose’ before commencing.

Materials

200g dry mineral samples

Standard reference materials (as supplied by your lecturer)

100mL beakers

100mL watch glasses

200mL volumetric flasks

Analytical balance

PPE

Spatulas

Concentrated Nitric acid (15M HNO_3)

Concentrated Hydrochloric acid (10M HCl)

Hot plate

Hot plate calibration device

Magnet

Prelab activity:

Reference standard details:

Reference standard No.	Reference standard name	Reference standard ID	Element	%
1			Fe	
2			Fe	
3			Fe	

Method

Sample Preparation

Note: Perform the analysis in duplicate

1. Weigh accurately (i.e. analytical balance) 1.0g of dried finely ground, into a 125mL conical beaker. If these are not available, a 100mL standard Pyrex beaker will suffice.

2. Weigh accurately 0.5g of a reference standard (as supplied by your lecturer) and record the relevant details in the table provided. Refer back to this table when comparing your results with those expected in the standard.

3. Treat the samples and reference standard by gently heating with 5mL hydrochloric acid in a covered 100mL beaker; do not boil.

4. When reaction has subsided, add 15mL concentrated (15M) nitric acid and evaporate to moist salts. Add 5mL concentrated (10M) hydrochloric acid and approximately 5mL deionised water.

5. Check any black residue that may be present for magnetite.

6. Warm gently to dissolve the salts.

7. Cool, quantitatively transfer to a 250mL volumetric flask and make up to volume using de-ionised water.

Calibration Standard Preparation

(Depending on time, the following standard preparation procedure may be performed by your supervisor)

8. Prepare fresh calibration standards containing 0-500ppm Fe.

9. The calibration solutions must contain HCl (~5%) to ensure solution preservation.

10. Label and store all solutions in plastic containers and store in a cupboard until required.

11. Your supervisor will provide either a stock solution from which you are to prepare your working solution or the pure metal from which a 1000ppm solution must be prepared. Check your workings with the supervisor before preparation of the calibration solutions.

Results

Magnetite check

Sample Identification	Test/Observation	Magnetite present (Y/N)

AAS Results: Iron analysis/Aqua Regia method

	Sample	Sample mass (g)	Volume (mL)	AAS Reading	Dilution	AAS Reading	ppm Fe	% Fe
1	Blank	NA	250		1			
2			250					
3			250					
4			250					
5			250					
6			250					
7			250					
8			250					
9			250					
10	Ref. Std.ID:		250					
11	Ref. Std.ID:		250					
12	Ref. Std.ID:		250					

Calculations

ppm Fe = (AAS reading x Volume x Dilution (if required))/Sample mass (g)

%Fe = (ppm Fe)/10000

Use the formula above to show how the final concentration of gold was calculated. Use the space provided:

% error:

Determine the % error of the result obtained from the reference standard, using the following formula:

% error = [(Experimental result – True result)/ (True result)] * 100

Use the space provided:

Questions

1. How is it possible to determine if your samples contained the mineral magnetite?

2. What method is appropriate if your sample contains magnetite and iron is to be determined?

3. Draw a table comparing the iron values obtained using AN200A and AN140A. Which of the two methods gives the higher result? Explain your answer.

Elements of Competency		**Assessor's Comments**
Interpret and schedule client requirements		
Prepare client samples sample for analysis		
Use (non) destructive methods to prepare laboratory portions for analysis		
Dispose of waste and spent samples		
Maintain a safe work environment		
I confirm that the above is my work and the assessor has given me sufficient feedback. **Candidate's signature:**		**Date:**
Candidate's comments:		
Assessor's signature:		**Date:**

Exercise 7: Method AN200A - Sodium Peroxide Fusion for Acid Insoluble Analytes

Introduction

Once samples have been sorted, crushed, split and pulverised, they are ready for the next part in the analytical process. Sample treatment can involve a number of processes depending on the mineral type being analysed and/or the analyte being sought in the sample. In this exercise, the samples prepared in the previous week will be treated in a number of different ways depending on the analyte being determined. Note the client job sheets for analytes requested and preparation methods required.

Aim

To use the sample preparation technique of fusion with sodium peroxide for the determination of analytes in a mineral sample.

Background

Some mineral types don't respond well to conventional acid digestion techniques. These can include magnetite, chromites, and ilmenite amongst others. These minerals can sometimes be referred to as 'refractory'. It is common to have an analyte in a sample only partially dissolved by acid, leaving more of the same analyte behind unaffected by the acid solution. In situations like this an alternative method is sought to completely bring the analyte into solution. In this exercise, the sodium peroxide fusion technique will be used to determine the amount of iron present in an iron ore sample.

Safety

High temperatures used in this practical. Avoid breathing fumes and use extraction hoods wherever possible.

Caution: Sodium Peroxide and concentrated Hydrochloric acid used in this exercise. Read MSDS before commencing the practical work. Wear gloves, lab coat and safety glasses and carry out digestion procedures in fume hoods provided. Ensure all PPE is 'fit for purpose' before commencing.

Materials

200g dry mineral samples

250mL beakers

250mL watch glasses

Analytical balance

Furnace (680-700º C)

Hot plates

Hot plate calibration device

PPE

Spatulas

Concentrated Hydrochloric acid (10M HCl)

Sodium Peroxide (Na_2O_2)

Zirconium crucibles

Reference standard details:

Reference standard	Reference standard ID	Element	%
1		Fe	
2		Fe	
3		Fe	

Method

4. Weigh between 0.3000 – 0.3500 g sample in duplicate into zirconium crucibles.

5. Place in muffle furnace for 30 minutes (600 º C) to remove sulphur and other volatile materials in the sample. Leave furnace door open slightly to assist oxidation of sulphur.

6. Take out, but do not allow to cool to ambient temperature as absorption of moisture can occur, thus making mixing of peroxide difficult. Cover sample with Na_2O_2 and stir well.

7. Set muffle at 670ºC.

8. Place samples into muffle for 15 to 20 minutes.

9. Take out and allow to cool slightly.

10. Place crucibles on side into beakers (250 mL) and add d.i. water to 250 mL mark.

11. Place watch glass on top.

12. Allow reaction to subside.

13. Add 35mL concentrated HCl. (Note: PPE to be worn!)

14. Allow to sit for ½ hour

15. Quantitatively remove crucible.

16. Place crucible and beaker on hot plate and heat to near boiling. This helps precipitate Zr as ZrO_2, thus clarifying the solution prior to analysis.

17. Quantitatively transfer to 500 mL volumetric flasks if using an instrumental technique to finish the determination.

Analysis

18. Read samples using AAS/ICP and estimate the amount of Fe in the sample against prepared standards.

19. If necessary, carry out dilutions to bring samples into analytical range.

Results

AAS Results: Fusion method

	Sample	Sample mass (g)	Volume	AAS Reading	Dilution	AAS Reading	ppm Fe	% Fe
1	Blank							
2								
3								
4								
5								
6								
7								
8	Ref. Std 1							
9	Ref. Std 2							
10	Ref. Std 3							

Calculations

ppm Fe = (AAS reading x Volume x Dilution (if required))/Sample mass (g)

%Fe = (ppm Fe)/10000

Use the formula above to show how the final concentration of iron was calculated. Use the space provided:

% error:

Determine the % error of the result obtained from the reference standard, using the following formula:

% error = [(Experimental result – True result)/ (True result)] * 100

Use the space provided:

Questions:

1. What are the main hazards in using sodium peroxide?

2. What disposal procedures need to be followed when dealing with a sodium peroxide spill?

3. Explain the importance of knowing a sample’s origin/history prior to testing/analysis?

Elements of Competency		**Assessor’s Comments**
Interpret and schedule client requirements		
Prepare client samples sample for analysis		
Use (non) destructive methods to prepare laboratory portions for analysis		
Dispose of waste and spent samples		
Maintain a safe work environment		
I confirm that the above is my work and the assessor has given me sufficient feedback. **Candidate’s signature:**		**Date:**
Candidate’s comments:		
Assessor’s signature:		**Date:**

Resources

Csuros, M. 1994, *Environmental sampling and analysis for technicians*, Lewis Publishers, Boca Raton, FL.
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Grader, F. E., 1990, *Sampling techniques – minerals*, Trust Publication, Perth WA. [East Perth Campus-Call Number 549.1 GRA]

Websites

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http://www.ltav.org.au/index.html - Laboratory Technicians' Association of Victoria

Wikipedia